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The Use of Sodium Per-Oxide for the Decomposition of Certain Ores and Industrial Products

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THE USE OF SODIUM PER-OXIDE

FOR THE

DECOMPOSITION OF CERTAIN ORES

AND

INDUSTRIAL PRODUCTS

BY

HERMAN ALBERT SCHOLZ

THESIS

FOR THE

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IN

CHEMISTRY

COLLEGE OF SCIENCE

UNIVERSITY OF ILLINOIS

PRESENTED JUNE, 1907

UNIVERSITY OF ILLINOIS

May 31 1907.

THIS IS TO CERTIFY THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

Herman Albert Scholz.

ENTITLED The Use of Sodium Per-Oxide for the Decomposition of Certain Ores and Industrial Products.

IS APPROVED BY ME AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE DEGREE OF Bachelor of Science in Chemistry.

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102010

THE USE OF SODIUM PEROXIDE FOR THE DECOMPOSITION OF CERTAIN ORES AND INDUSTRIAL PRODUCTS.

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THE USE OF SODIUM PEROXIDE FOR THE DECOMPOSITION OF CERTAIN ORES AND INDUSTRIAL PRODUCTS.

Historical.

Sodium peroxide was first used in analysis as a fusion medium in 1893.[1] At that time J. Clark used it for decomposing pyrites, zinc blende, galena, and chrome and arsenic ores. He cautiously heated the ore with six times its weight of sodium peroxide in an open nickel or platinum crucible. He states that the action of sodium peroxide on coke and coal is too violent for analytical purposes. W. Hempel[2] employed sodium peroxide for the oxidation of chromium, manganese, tungsten, and titanium-iron ores. He used from two to four times as much sodium peroxide as ore, and made the fusions in a platinum or silver crucible. He states that sulphides are oxidized with explosive force and for their determination he used one part of sample to two parts of sodium carbonate and four parts of sodium peroxide. He also mentions that sulphur is completely oxidized to sulphur trioxide.

Spiller and Kalman[3] used sodium peroxide on ferro chrome, chrome steel, chrome iron ore and sulphides.

A. Edinger[4] heated organic substances with a mixture of sodium peroxide and sodium carbonate in a nickel or a porcelain crucible in order to make chlorine and sulphur determinations. E. H. Saniter[5] decomposed chrome ore and chrome iron ore by heating the ore with about six times its weight

of sodium peroxide in a nickel crucible.[6] C. Glaser used sodium peroxide to determine sulphur in coke, coal and asphalt. He placed the substance into a nickel or a silver dish, covered it with four times its weight of sodium carbonate, and upon that placed a piece of sodium hydroxide weighing about twice as much as the sample taken. The whole mass was heated carefully until the reaction ceased and then sodium peroxide was carefully sifted into the crucible until the substance was entirely decomposed.[7] Henri Angenot used sodium peroxide for decomposing tin, antimony, and arsenic ores and alloys. He heated 1/2 gm. of the substance with 7.0 gm. sodium peroxide in an iron crucible.[8] Louis Lucchese used a mixture of equal parts of sodium peroxide and sodium carbonate in a platinum crucible for decomposing certain iron-silicon compounds.

S. W. Parr[9] made fusions of coal with sodium peroxide in a closed steel or a brass crucible in his calorimeter. He used one part of coal to seventeen of sodium peroxide for the best results. In this fusion, after starting the reaction by means of a red hot iron wire, the combustion proceeds without the addition of further heat. Enough heat is generated to carry out the reaction and fuse the mass. He also found that the sulphur in the coal was entirely oxidized to sulphur trioxide.[10] He later found that with anthracite coals it was necessary to use an accelerator in the shape of a free burning substance such as sugar, tartaric acid, etc., and a superoxide

such as barium peroxide, potassium chlorate, or potassium persulphate. The most intense oxidizing effect could be obtained by the use of a mixture of 1.0 gm. potassium persulphate, 0.5 gm. tartaric acid to 12 gm. sodium peroxide and 0.5 gm. coal.

For the determination of the halogens in organic compounds H. H. Pringsheim[11] mixed the substance with 16 to 18 times its weight of sodium peroxide and ignited in a covered iron crucible by passing a red hot wire through a hole in the cover.[12] He also gives a general method for the use of sodium peroxide in qualitative analysis to detect halogens, phosphorus, arsenic, and sulphur. Sodium peroxide is mixed with 1/25 of its weight of a free burning, non-hygroscopic substance such as naphthalene. The substance to be tested is added in an iron crucible which is heated until the reaction begins. He reports quantitative determinations of arsenic and phosphorus[13] in organic compounds. He found it necessary to add sugar when the substance contained less than fifty per cent of carbon and hydrogen. His latest communications concerning the use of sodium peroxide in organic quantitative analysis were published in 1906.[14]

For making qualitative analyses of organic substances, Von Konek used a cylindrical nickel or a steel crucible similar to that designed for Parr's calorimeter.[9] The finely powdered sample was mixed with 5 to 10 gm. sodium peroxide and a small amount of a compound rich in carbon, such as camphor. The

mass was ignited by dropping in a red hot iron wire. The reaction was violent. He states that the mass contained the carbon as carbon dioxide, the nitrogen as nitric acid, the sulphur as the trioxide, the phosphorus as the pentoxide, and the halogens as oxygen compounds. Von Konek and Zöhls[16] report that by mixing flour with sodium peroxide, potassium persulphate, and tartaric acid in a steel cylinder and starting the reaction as above, all the nitrogen was oxidized to nitric acid and could be quantitatively determined as such.

Object.

From the above brief historical resume it may be seen that while sodium peroxide has been used as a fusion medium for certain substances, it has always been by heating a mixture of the ore and sodium peroxide in an open crucible over a free flame. This does not always give as complete a decomposition of the substance as is desirable and usually takes a long time. Moreover it attacks the crucibles, and introduces a considerable amount of foreign substance into the determination.

The reaction between sodium peroxide, a combustible substance, and an accelerator or intensifier has never been used for the purpose of decomposing inorganic compounds of this sort, although Parr and later Pringsheim and Von Konek used it successfully for organic compounds. It was therefore decided to make some experiments along this line and, if possible, develope a method for fusing certain ores, glazes, etc.,

for which there is at present no rapid means of decomposition. Quantitative tests were made on the following substances: a lead glaze; a lead slag; a manganese glaze; a franklinite ore; a sample of ferro manganese and a blast furnace slag (for SiO_2).

Qualitative experiments were run on Bauxite, Monazite, a fire clay, a brick clay, and a chrome iron ore.

Reagents used.

The sodium peroxide used in these experiments was the ordinary commercial article made by Koenig and Co. It contained traces of SiO_2, Al_2O_3 and Fe_2O_3. The amount of sodium peroxide was determined by measuring the volume of oxygen given off from a weighed amount of the substance when decomposed by water containing a few drops of cobalt nitrate.[17] Owing to the difficulty of obtaining a fair sample and the great rapidity with which the sodium peroxide was changed by the moisture of the atmosphere, the results obtained were only approximate. They showed that there was between 95% and 96% Na_2O_2 in the commercial product.

Ordinary so called "C. P." zinc sulphide was used. It was found to be free from lead and manganese and contained only very slight traces of chlorides. It was rather moist, and before being used was dried at 110° C. for an hour.

The pyrite (FeS_2) used was the purest specimen of the mineral which could be obtained. It was finely ground

and passed through a 200 mesh sieve. On testing it was found to be free from manganese.

The other reagents used were all of standard purity and where there was any doubt they were first tested.

Experimental.

(a) Analysis of the lead ores.

The ores used in the experiments, containing about 65% and 71% of galena respectively, were analyzed for lead by means of the standard method. 0.5 gm. of the finely powdered ore (through 200 mesh sieve) was boiled in a covered casserole with 25 cc. concentrated nitric acid (1.42 sp. gv.) to which a few drops of hydrochloric acid had been added. The boiling was found to be necessary for in one case, when the sample was boiled only half an hour the results obtained were several per cent low. After the action of the nitric acid had ceased, sulphuric acid (sp. gv. 1.82) was added and the solution boiled down to sulphuric acid fumes. Water was added to the cool solution, which was then boiled to get all the iron into solution. Enough water and alcohol were added to make a 1% sulphuric acid and 10% alcohol solution. After cooling the clear solution was decanted through a filter, retaining as much of the residue as possible in the casserole. This was thoroughly washed with a 1% sulphuric acid and 10% alcohol solution. The lead sulphate was dissolved in hot ammonium acetate solution by boiling for 20 minutes. The silica, etc.,

was filtered off and washed with hot water. The lead sulphate was reprecipitated by means of dilute sulphuric acid, enough alcohol was added to make a 10% solution, and the precipitate was allowed to settle. It was filtered on a previously prepared Gooch crucible, washed with a 1% sulphuric acid and 10% alcohol solution and finally with alcohol until free from sulphuric acid. It was dried at 110° C. for an hour, and weighed. From the weight of $PbSO_4$ the per cent of lead was calculated. Ore No. 5 ran 61.58% and 61.62% Pb. It had little pyrites and very little silica. Ore No. 22 ran 56.05% and 55.98% Pb. It had considerable pyrites and silica and a little manganese.

(b) Fusion of lead ore with sodium peroxide in an iron crucible.

0.3 gm. ore No. 5 (200 mesh sieve) was mixed with 5.0 gm. sodium peroxide in an iron crucible and heated with a free flame until the mass was molten. It was rotated to prevent the settling of the ore. It was kept in a molten condition for five minutes, cooled, taken up in water, made neutral with sulphuric acid and enough excess of acid added to make a 1% sulphuric acid solution. Sodium bisulphite was added to reduce the lead peroxide formed in the fusion, and the solution was boiled. Enough alcohol to make a 10% solution was added and the lead was determined gravimetrically as lead sulphate. The results obtained gave 60.06% and 60.07% Pb with ore, No.5, which contains 61.60% Pb. The

low results were due to incomplete decompostion, or to the interference of the iron in keeping the lead sulphate from dissolving in the ammonium acetate solution.

(c) Experiments to completely decompose lead ores.

Further tests were made with ores No. 5 and No. 22.

Experiment No. 1.

0.5 gm. of the finely powdered ore (through 200 mesh sieve) was mixed with 5.0 gm. sodium peroxide in a closed brass cylinder or bomb such as is used in Parr's calorimeter.[9] The mixture could not be ignited by means of a red hot iron wire. When 0.2 gm. powdered magnesium had been added the mixture went off but it sintered rather than fused and did not completely decompose the ore. The amounts and proportions of the different constituents were varied, and boric acid, varying in amount from 0.2 to 0.5 gm. was added but no complete decomposition was obtained.

Experiment No. 2.

Added finely powdered sugar to the mixtures in varying proportions with and without magnesium, and with and without boric acid. The sugar was later replaced by starch, and it was found that an apparently complete decomposition of the ore was obtained by the following mixture: 7.0 gm. sodium peroxide, 0.5 gm. ore No. 22, 0.3 gm. starch. The fusion was a pasty mass and the reaction lasted 15 to 20 seconds.

Clark[1] states that in galena fusions with sodium peroxide the lead was present partly as insoluble lead peroxide and partly in a soluble form, probably as sodium plumbate. It was therefore resolved to find out in what form the lead was present, and to get it all as lead peroxide, if possible, when it could be determined directly by some of the volumetric methods for determining lead peroxide. The fusion from the last named mixture was taken up in about 100 cc. of cold water. It was made exactly neutral to litmus paper with strong acetic acid[18] to render insoluble any sodium plumbate, which is soluble in a strongly alkaline solution and decomposed in an acid solution, but which is insoluble in a neutral solution. It was filtered without boiling. On testing with potassium dichromate in an acetic acid solution much lead was found in the filtrate. This shows that some of the lead was probably present as carbonate which was dissolved by the acetic acid.

(d) Experiments to change the lead in ores to the peroxide.

Experiments were made to obtain a mixture which after fusion would leave all the lead as peroxide, or at least in a form insoluble in a neutral solution.

Experiment No. 3.

It was found that a mixture of 8.0 gm. sodium peroxide, 0.5 gm. ore No. 22, and 1.0 gm. ammonium carbonate gave

a fusion in which the ore was apparently completely decomposed. When taken up in 100 cc. of cold water, made exactly neutral with acetic acid and filtered without boiling, there was no lead in the filtrate. This fusion spattered considerably on the top and sides of the bomb and there was a certain amount of pressure due to the ammonia and nitrogen gas liberated.

It was found that a mixture in which the ammonium carbonate was replaced by 1.0 gm. ammonium oxalate gave practically the same results. Since there was manganese in ore No. 22, lead dioxide could not be determined in it directly. Ore No. 5 which contained no manganese was therefore used for the quantitative experiments. The above ammonium carbonate mixture did not completely decompose No. 5, but the oxalate mixture apparently did. It gave a fusion in which all the lead remained in an insoluble state after the aqueous solution had been made exactly neutral with acetic acid.

Experiment No. 4.

For quantitative experiments a closed steel bomb was employed like that used in the first experiments exeept that the charge was ignited by an electrically heated iron wire which dipped down into the fusion mixture. A mixture of 8.0 gm. sodium peroxide, 0.5 gm. ore No. 5 (200 mesh wire), 0.8 gm. ammonium oxalate was thoroughly mixed in the crucible, placed in a can of water and ignited by passing an electric

current through the fine wire. The fused mass was removed from the crucible, taken up in 100 cc. of cold water, made exactly neutral with acetic acid, filtered on a Hirsch funnel covered with asbestos, and washed two or three times with hot water. There was no lead in the filtrate. The mass of asbestos, PbO_2, etc., was placed in a beaker and the lead peroxide determined by the method of Toff[19], which depends on the equation: $PbO_2 + 4HI = PbI_2 + I_2 + 2H_2O$. The lead peroxide was treated with 1.2 gms. potassium iodide and ten times that amount of sodium acetate in 55 cc. of 50% acetic acid solution. The solution was diluted to 250 cc. and titrated with a sodium thiosulphate solution that had been standardized against a known weight of pure potassium bromate. The amount of lead as PbO_2 found in ore No. 5 (containing 61.60% Pb) was 34.29% and 42.04%Pb or an average of less than 2/3 of the lead in the ore was present as the peroxide.

The lead peroxide was determined by means of a standard oxalic acid solution in the presence of sulphuric acid. The excess of oxalic acid was titrated against a standard potassium permanganate solution.

$$(COOH)_2 + PbO_2 + H_2SO_4 = PbSO_4 + 2H_2O + 2CO_2$$

The solution of potassium permanganate was standardized against a weighed amount of Kahlbaum's chemically pure ferrous ammonium sulphate and the oxalic acid solution standardized against the permanganate.

Experiment No. 5.

A mixture of 7.0 gm. sodium peroxide, 0.5 gm. ore No. 5, and 1.0 gm. ammonium oxalate was fused as before. It was taken up in water, made exactly neutral with acetic acid, and filtered on a Hirsch funnel. The asbestos and precipitate were removed to a beaker, 50 cc. of the standard oxalic acid solution were run in from a pipette and sulphuric acid was added in decided excess. The solution was boiled for five minutes and while still hot the excess of oxalic acid was determined by titration against the standard permanganate. In this way the amount of oxalic acid used in reducing the lead peroxide could be determined and consequently the amount of lead. The lead found in ore No. 5 was 43.61% and 38.37%Pb. A little over 2/3 of the lead was present as the peroxide.

Experiment No. 6.

Experiments were continued to find, if possible, a mixture which would convert all the lead into lead peroxide. A mixture of 7.1 gm. sodium peroxide, 0.5 gm. ore No. 5, and 1.0 gm. ammonium chloride fused in the closed bomb gave an apparently complete fusion. The lead was all rendered insoluble by making the solution exactly neutral with acetic acid. This insoluble precipitate was filtered off. There was no lead in the filtrate. The precipitate was washed with water until free from all traces of chlorides and sulphates. Part of the precipitate was boiled vigorously with water and

then quickly filtered while hot. The filtrate was found to be free from both lead and chlorides. This showed that no lead chloride was present. Another portion of the precipitate was boiled for 15 minutes with a strong ammonium acetate solution. It was filtered and the filtrate tested with HCl and $BaCl_2$ for sulphates, but no precipitate of barium sulphate was found. This showed that no lead sulphate was present. Repeated the experiments with fresh portions and obtained the same results.

Quantitative experiments with this mixture, in which the lead peroxide was determined by means of oxalic acid as above, gave in ore No. 5, 54.13% and 47.48%Pb present as peroxide or about 80% of the total lead. This was the highest per cent of lead found as lead peroxide in any of the experiments. A mixture of 5.0 gm. sodium peroxide, 0.3 gm. ore No. 5, and 1.0 gm. ammonium chloride gave 34.63% and 44.31%Pb as peroxide.

(e) Attempts to change lead to peroxide by addition of sodium peroxide to the solution after the fusion.

Experiment No. 7.

Fusions of the last named mixture were taken up in water and three or four grams of sodium peroxide were slowly added with much stirring. The whole was boiled, made neutral with sulphuric acid, and phosphoric acid and manganese sul-

phate added to prevent the injurious effects of any chlorides present. Twenty-five cc. of standard oxalic acid were run in, it was made strongly acid with sulphuric acid and boiled. The excess of oxalic acid was titrated with permanganate and the lead present as lead peroxide calculated. The results for ore No. 5 were 52.68% and 38.03%Pb.

In the foregoing experiments the attempt to convert all the lead to lead peroxide was unsuccessful. The lead present as peroxide varied from 65% to 80% of the total lead. No lead chloride or lead sulphate could be found in the precipitate and in the NH_4Cl fusions no carbon compounds were present. The lead which did not exist as peroxide was therefore probably present as other oxides of lead. As it did not seem worth while to make further attempts to convert all the lead to lead peroxide, it was decided to use a mixture that would decompose the ore and leave the resulting fused mass in such a form that the lead could be easily changed to lead sulphate and filtered off. The ammonium chloride mixture was out of the question because it introduced hydrochloric acid which was not desirable. The ammonium oxalate mixture, while it gave an apparently complete decomposition, spattered a great deal and made considerable pressure in the bomb. The starch mixtures were therefore employed in the first experiments.

(f) Sodium peroxide fusions, using starch as the combustible material.

Influence of an accelerator.

It was found that by adding an accelerator in the form of potassium persulphate to the sodium peroxide and starch a better fusion and apparently complete decomposition was obtained.

Experiment No. 8.

5.0 gm. sodium peroxide.

0.3 gm. ore No. 5 (200 mesh sieve).

0.2 gm. starch.

0.3 gm. potassium persulphate.

The above mixture was ignited in an electric bomb, the melt dissolved in 100 cc. of water in a 4 inch casserole, the sodium hydroxide neutralized with sulphuric acid (1.42 sp. gv.) and enough of the acid added to make a 2% excess. Sodium bisulphite was added to reduce the lead peroxide and the solution was boiled for 5 minutes. It was diluted to 200 cc. and enough alcohol added to make a 10% solution. Continued the determination of the lead gravimetrically as the sulphate as before. The following results were obtained with ore No. 5, 57.49%, 60.22%, and 59.13%Pb.

(g) Sodium peroxide fusions, using zinc sulphide for the combustible material.

Since the fusions with starch did not give satisfactory results, experiments were made in which the heat for the

decomposition was furnished by the oxidation of zinc sulphide.

Experiment No. 9.

8.0 gm. sodium peroxide.

0.3 gm. potassium persulphate.

0.8 gm. zinc sulphide.

0.5 gm. ore No. 5.

The above mixture was ignited by means of a red hot iron wire. The fusion was liquid and red hot and the decomposition appeared complete for the entire melt was dissolved in nitric acid leaving no residue. It was found the combustion took place better in an open bomb than in one which was tightly closed. The reaction was instantaneous and the fusion quiet and without any sputtering. Quantitative experiments with the above mixture in an open bomb gave for No. 5, 60.61% and 59.73%Pb, results which were slightly better than those obtained in the starch fusions.

The above results were obtained by determining the lead by the Molybdate Method[20]. The melt from the fusion was treated just as given on the previous page, up to the point where the lead sulphate was dissolved in ammonium acetate solution. This solution of lead sulphate in ammonium acetate was diluted to 250 cc., made slightly acid with acetic acid and heated to boiling. The boiling hot solution was titrated with a standard solution of ammonium molybdate. A solution of 1 gm. tannin in 300 cc. of water was used as an outside

indicator. 8.0 gms. of molybdic acid were dissolved in the required amount of ammonium hydroxide (0.96 sp. gv.) and made up to 1000 cc. A weighed amount of lead sulphate, which had been prepared chemically pure in the determination of lead as lead sulphate, was dissolved in a hot ammonium acetate solution. It was made acid with acetic acid, diluted to 250 cc., and heated to boiling. Then molybdate solution was run in until the point was reached where the addition of one drop of the molybdate gave a solution one drop of which colored yellow a drop of tannic acid solution placed on a titration plate. The appearance of the yellow color served to indicate the end point of the reaction. A certain amount of the molybdate solution was required to produce the yellow color. This amount was determined by measuring the amount of molybdate required to produce the yellow color in a solution like the above, but without any lead being present. This blank was subtracted from each subsequent titration.

Fusions of ore No. 22 were made with the same mixture as above.

Experiment No. 10.

8.0 gm. sodium peroxide.

0.3 gm KSO_4.

0.8 gm. zinc sulphide.

0.5 gm. ore No. 22 (200 mesh sieve).

Ignited the above mixture with a red hot iron wire. The fusion was good and the decomposition of the ore appeared

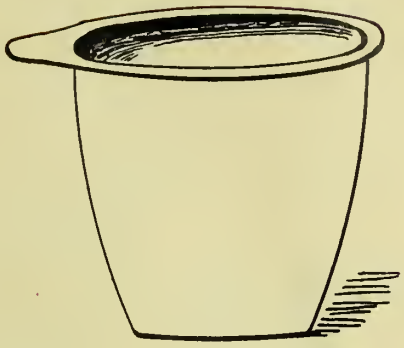
complete. Determined by the Molybdate method, and obtained the following results: 55.51%, 55.18%, 55.45%, and 55.13%Pb. The ore contained 56.01%Pb.

The fusions with zinc sulphide gave results which were low but which were better than those with starch. The fusions were so hot and the melt so fluid that it appeared possible that something else was the cause of the incomplete decomposition. The shape of the bomb and the fact that it had to be water cooled were factors which might have hindered complete decomposition. Consequently it was decided to use a nickel crucible.

(h) Fusions in a nickel crucible.

It was thought possible that the above incomplete decompositions might have been due to the fact that a slight amount of the ore had lodged in the sharp lower corner of the bomb or around its water cooled side and was consequently insufficiently heated. Since the fusions took place quietly and without sputtering it was decided to use a nickel crucible.

This did not have to be water cooled and thus gave hotter fusions. It had rounded corners and afforded no place for the ore to lodge. It was smaller than the bomb and consequently the melt could be more easily removed and less water needed for solution. The nickel crucible used was of the size and shape shown in the drawing on next page, and had a capacity of 35 cc.



NICKEL CRUCIBLE
Actual Size

Since the iron wire used for the ignition caused considerable trouble by the formation of difficultly soluble magnetic oxide of iron, it was replaced by magnesium ribbon. Moreover it is advisable to keep the quantity of iron salts as low as possible in lead determinations, for they interfere as will be shown later.

Experiment No. 11.

8.0 gm. Na_2O_2.

0.3 gm. KSO_4.

0.8 gm. ZnS.

0.5 gm. ore No. 5 (200 mesh sieve).

The sodium peroxide was put into the crucible by means of a small aluminum measure. The other substances were added and carefully and thoroughly mixed with a glass rod, which was then brushed clean. The cover was put on the

crucible, and was moved far enough to one side to permit the dropping in of a 1/2 to 3/4 inch piece of lighted magnesium ribbon. After the ignition had taken place it was immediately replaced. The fusion was very quiet and did not spatter to any appreciable extent.

The lead determined by the molybdate method gave for ore No. 5, 61.23% and 61.22% Pb, and for ore No. 22, 55.82% and 55.68% Pb. These results were better than any which had yet been obtained, but were not entirely satisfactory. The trouble was due either to incomplete decomposition of the ore or to complications caused by the presence of some other substance, probably iron.

Four more fusions of No. 5 were made in the same way and the lead determined gravimetrically as sulphate. The results were even lower, viz., I. 60.44%, II. 59.07%, III. 60.68%, and IV. 60.64% Pb. In all of these the residue after taking up in water, acidifying with sulphuric acid, adding sodium bisulphite, and boiling for 5 minutes was of a browner color than before, this being particularly noticeable in the case of determination II. This brown color was no doubt due to basic sulphates of iron which are precipitated upon boiling even in a slightly acid solution in the presence of large quantities of alkali salts.[21] These basic iron salts apparently keep some of the lead sulphate from being dissolved by the ammonium acetate solution. Their effect is similar to that produced by the presence of $BaSO_4$ and silica.[22] The ammonium acetate solution of lead was found on testing to contain but very slight traces of iron.

(i) Experiments to prevent the contamination of $PbSO_4$ with basic iron salts.

Experiment No. 12.

Fusions of No. 5 were made and the melt treated as before except that the boiling on acidifying was omitted. The solution was heated just to boiling for a moment and then allowed to cool. The residue still had a slight brown color but it was much lighter than before. Determined by the molybdate method, the results were: 61.23% and 61.16%Pb, which was still low. Apparently when the melt was dissolved some iron hydrate was formed which did not dissolve in the dilute acid.

Experiment No. 13.

Fusions of No. 5 made as before. The crucible and melt were put into a casserole and about 75 cc. of water containing 7 cc. sulphuric acid (1.84 sp. gv.) added, (enough acid to almost neutralize exactly all the sodium hydroxide present). The melt dissolved in two or three minutes and the crucible was immediately taken out before the acid had a chance to attack the nickel. An excess of 2 cc. sulphuric acid (1.84 sp. gv.) was added and a concentrated solution of sodium bisulphite slowly poured in with much stirring. The solution was heated to boiling for a moment, diluted to 200 cc. and cooled. 20 cc. of alcohol were added. The residue

was almost pure white. The lead determined by the molybdate method gave 61.46%Pb. Repeated exactly as before and got 61.51% and 61.57%Pb. These results agree with the amount of lead present: 61.60%Pb.

Exactly the same method applied to ore No. 22 gave 55.89%, and 55.88%Pb. This ore contains: 56.01%Pb.

The following table shows the results obtained in the foregoing experiments;

TABLE NO. 1.

Ore No. 22. (Galena Ore).

Method of Decomposition.	Method of Determination.	%Pb found.	Remarks.
Standard Method. HNO_3 + HCl	Gravimetrically as $PbSO_4$.	56.04 55.98	
Na_2O_2, KSO_4, ZnS in bomb.	Molybdate Method.	55.51 55.18	
$Na_2O_2KSO_4$, ZnS in bomb	" "	55.45 55.13	
Na_2O_2, KSO_4, ZnS in Ni Crucible	" "	55.82 55.68	Low results probably due to iron.
Na_2O_2, KSO_4, ZnS in Ni Crucible	" "	55.89 55.90 55.88	Melt taken up in acidified water and solution not boiled

Ore No. 5. (Galena Ore).

Method of Decomposition.	Method of Determination.	% Pb found.	Remarks.
Standard Method. HNO_3 + HCl	Gravimetrically as $PbSO_4$	61.58 61.62	
Na_2O_2 heated in iron crucible.	Gravimetrically as $PbSO_4$	60.06 60.06	Low results probably due to iron.
Na_2O_2, KSO_4 & starch in bomb.	Gravimetrically as $PbSO_4$	60.22 59.13	
Na_2O_2, KSO_4 & ZnS in bomb.	Molybdate Method.	61.36 60.47	
Na_2O_2, KSO_4 & ZnS in Ni Crucible	" "	61.23 61.23	
Na_2O_2, KSO_4 & ZnS in Ni Crucible	Gravimetrically as $PbSO_4$	60.44 59.07 60.68 60.64	Residue was brown, due to basic iron salts.
Na_2O_2, KSO_4 & ZnS in Ni Crucible.	Molybdate Method.	61.23 61.16	The solution was not boiled.
Na_2O_2, KSO_4 & ZnS in Ni crucible.	" "	61.46 61.51 61.57	Melt taken up in acidified water and solution not boiled

(k) Experiments to determine application of above method as a general method for lead determinations.

The foregoing experiments suggested the possibilty of using the sodium peroxide zinc sulphide fusion as a general method for decomposing substances in which lead is to be determined. In order to test this idea the following substances were analized:

1. A galena lead ore, No. 45, high in pyrites and silica, but low in lead. It was decomposed by boiling a 1.0 gm. sample (through 200 mesh sieve) for 40 to 50 minutes in nitric acid(1.12 sp. gv.) according to the method given on page 6. The lead was determined gravimetrically as the sulphate. Found 3.13% Pb.

Experiment No. 14.

10.0 gm. sodium peroxide.
0.8 gm. zinc sulphide.
1.0 gm. ore No. 45 (200 mesh sieve).
0.3 gm. potassium persulphate.

The above mixture was ignited by a lighted magnesium ribbon in the usual manner. The fusion was very hot as was shown by the bright red color to which the crucible was heated. This great heat was probably due to the burning of the pyrites. The melt was treated as before and the lead determined by the Molybdate method.
Results: 3.16% and 3.19%Pb.

2. A lead glaze, high in calcium and silica. 1.0 gm. sample (200 mesh sieve) was decomposed by the nitric acid method and the lead determined by the molybdate method. 27.81% and 27.82% of lead was found in the sample. The great amount of silica present caused difficulties both in filtering the insoluble residue and in dissolving the lead sulphate in ammonium acetate solution, necessitating a much longer boiling.

Experiment No. 15.

10.0 gm sodium peroxide.
1.5 gm. zinc sulphide.
0.4 gm. KSO_4.
1.0 gm. glaze (200 mesh sieve).

Since there was no combustible matter in the sample it was necessary to raise the proportion of zinc sulphide. The fusion was good and the glaze apparently completely decomposed. The melt was dissolved in water, the absence of iron making unnecessary the precautions taken in the previous analyses. The lead determined by the molybdate method gave 27.84% and 27.82% Pb. In this fusion the silica went into solution almost completely and was filtered off from the insoluble residue and thus did not interfere in the filtering nor in the solution of the lead sulphate.

3. A lead slag was analyzed. 0.5 gm. of the slag (100 mesh sieve) was decomposed by nitric acid and hydrochloric

acids and determined by the molybdate method. Result: 36.39% Pb.

Experiment No. 16.

8.0 gm. sodium peroxide.

1.2 gm. zinc sulphide.

0.3 gm. KSO_4.

0.5 gm. lead slag (100 mesh sieve).

Ignited with magnesium wire. The fusion was good and the decomposition appeared to be complete. Since there was very little iron present, the melt was dissolved in water, and treated in the usual manner and the lead determined by the molybdate method. Results were 36.58% and 36.48% Pb.

These results by the sodium peroxide method compare very favorably with those obtained by the standard nitric and hydrochloric acid decompositions, as may be better seen from the tabulated results.

Table No. 2.

Comparison of lead determinations by standard and by sodium peroxide methods of decomposition.

Sample.	Method of Decomposition.	Method of Determination.	% Pb.
Galena ore No.5.	Standard Method HNO_3 + HCl	Gravimetrically as $PbSO_4$.	61.58 61.62
" " No.5.	Na_2O_2, KSO_4, ZnS, in Ni crucible.	Molybdate method.	61.46 61.51 61.57
" " " 22.	Standard Method HNO_3 + HCl	Gravimetrically as $PbSO_4$.	56.04 55.98
" " " 22	Na_2O_2, KSO_4, ZnS in Ni crucible	Molybdate method.	55.89 55.90 55.88
" " " 45	Standard Method HNO_3 + HCl	Gravimetrically as $PbSO_4$.	3.13
" " " 45	$Na2O_2$, KSO_4, ZnS, in Ni crucible	Molybdate method.	3.16 3.19
Lead Glaze.	Standard method HNO_3 + HCl	" "	27.81 27.82
" "	Na_2O_2, KSO_4, ZnS in Ni crucible.	" "	27.84 27.88
Lead Slag.	Standard method HNO_3 + HCl	" "	36.39
" "	Na_2O_2, KSO_4, ZnS in NI crucible.	" "	36.48 36.58

(1) Sodium peroxide method of decomposition applied to manganese determinations.

Since the sodium peroxide and zinc sulphide fusions were so effective as fluxes in the lead determinations, it was decided to try the mixtures on manganese compounds.

1. Analysis of a Manganese glaze.

A manganese glaze high in silica but containing no calcium was first used. It was not attacked to any appreciable extent by nitric and hydrochloric acids, even after boiling for an hour. 1.0 gm. of the glaze (200 mesh sieve) was fused with sodium carbonate in a platinum crucible. The melt was dissolved, made acid with sulphuric acid (1.42 sp. gv.) and the manganese determined by Volhard's[23] method as given below. The acid solution was made almost neutral with sodium hydroxide and then a slight excess of an emulsion of zinc oxide added. It was diluted to 200 cc., 40 cc. of a 20% zinc sulphate solution added, heated to boiling, 2 or 3 drops of nitric acid (1.42 sp. gv.) added, and the boiling hot solution titrated with potassium permanganate solution until the clear supernatant liquid remained pink. The permanganate solution was standardized in the same way against a weighed amount of Kahlbaum's manganese sulphate, which was dried to constant weight by heating in a platinum dish to a dull red.

4.57% and 4.55% MnO found in the sample.

Experiment No. 17.

10.0 gm. sodium peroxide.

1.4 gm. zinc sulphide.

0.4 gm. KSO_4.

1.0 gm. manganese glaze (200 mesh sieve).

Ignited in a nickel crucible by means of magnesium ribbon. Very good fusion and apparently complete decomposition for no residue was left upon acidifying with sulphuric acid. The melt was dissolved in water, acidified with sulphuric acid (1.42 sp. gv.) and sodium bisulphite added to reduce the MnO_2. The sulphur dioxide was boiled off and the manganese determined by Volhard's method. Results: 4.57% and 4.55%MnO.

2. Analysis of Franklinite.

The sample of franklinite, (Zn,Fe) $(Fe_2Mn_2)O_4$, contained considerable silica. It is a very refractory mineral which can not easily be decomposed by sodium carbonate fusion, is decomposed with great difficulty by $KHSO_4$ fusion, and is decomposed by concentrated nitric and hydrochloric acids only after very long boiling. A 1.0 gm. sample (200 mesh sieve) was boiled for two hours with nitric acid (1.42 sp. gv.) containing a few drops of hydrochloric acid (1.12 sp. gv.). 10 cc. sulphuric acid (1.42 sp. gv.) were added and the solution boiled down to sulphuric acid fumes. It was diluted, nearly neutralized with sodium hydroxide, zinc oxide emulsion added until the iron was precipitated as hydroxide and a slight

excess of zinc oxide left. The solution was diluted to exactly 500 cc. and allowed to settle. Exactly 200 cc. of the clear supernatant liquid was decanted off and the manganese determined by the Volhard method. Results were 7.89% and 7.96% MnO.

Experiment No. 18.

8.0 gm. sodium peroxide.

1.2 gm. zinc sulphide.

0.4 gm. KSO_4.

1.0 gm. franklinite (200 mesh sieve).

Ignited by magnesium ribbon in nickel crucible. The fusion was liquid and red hot but the decomposition was not complete. The proportions of the components were varied but it was impossible to get a complete decomposition with these ingredients. It was there fore decided to substitute for the zinc sulphide some substance which would give more heat. Iron pyrite was found to answer this requirement very well.

Experiment No. 19.

8.0 gm. sodium peroxide.

0.2 gm. sulphur (pulverized).

0.3 gm. KSO_4.

2.0 gm iron pyrite.

1.0 gm franklinite (200 mesh sieve).

Ignited by magnesium wire in nickel crucible. The fusion was extremely hot and remained liquid and red hot for about two minutes. It gave a complete decomposition of the franklinite but some of the sulphur remained unoxidized. The melt dissolved very slowly, in about two hours. This was remedied by sifting 3 to 4 gm. of sodium peroxide into the liquid fusion and rotating the crucible, slightly, to mix the peroxide throughout the mass. The resultant melt dissolved easily in water in 3 to 4 minutes.

By using powdered magnesium a cleaner melt was obtained and there was no annoyance due to unburned sulphur.

Experiment No. 20.

8.0 gm. sodium peroxide.

0.3 gm. powdered magnesium.

0.3 gm. KSO_4.

2.0 gm. pyrites.

1.0 gm franklinite (200 mesh sieve).

Ignited by magnesium ribbon. 3to 4 gms. of sodium peroxide were added to the liquid fusion to make the melt go into solution easily. The solution was made acid with sulphuric acid (1.42 sp. gv.), the MnO_2 reduced with $NaHSO_3$ as before, and the manganese determined by Volhard's method as in the other franklinite determination. Found: 7.90% and 7.88% MnO.

3. Analysis of Ferro-manganese.

A 0.5 gm. sample of ferro-manganese which had been passed through a 100 mesh sieve, was dissolved by boiling for about an hour in nitric acid (1.42 sp. gv.). Sulphuric acid was added and the solution was evaporated to sulphuric acid fumes. The manganese was determined by Volhard's method as in the franklinite determinations. Results were 56.25% and 56.23% Mn.

Experiment No. 21.

8.0 gm. sodium peroxide.

0.5 gm ferro manganese (100 mesh sieve).

0.3 gm. magnesium.

0.3 gm. KSO_4.

Ignited by means of magnesium ribbon in the usual way. The reaction was slow and consisted of a series of sputterings which gave a sintered mass. The ferro-manganese was not completely decomposed.

Experiment No. 22.

8.0 gm. sodium peroxide.

0.5 gm. ferro-manganese (100 mesh sieve).

0.8 gm. zinc sulphide.

0.3 gm. KSO_4.

Ignited by means of magnesium ribbon. A red hot liquid fusion was obtained, which apparently decomposed the substance completely. The melt dissolved with difficulty.

3 to 4 gms. of sodium peroxide sprinkled into the liquid fusion produced a melt that was dissolved easily in a few minutes. The solution was acidified with sulphuric acid, the MnO_2 reduced with $NaHSO_3$, and the manganese determined by Volhard's method as before. Results: 56.18% and 56.32% Mn.

Table No. 3.

Comparison of Standard and Sodium peroxide methods of decomposition for Manganese determinations.

Sample	Method of Decomposition.	% obtained.
Manganese glaze.	Standard Method.	4.57% MnO.
	Na_2CO_3 fusion.	4.55% MnO.
Manganese glaze.	Na_2O_2, ZnS, KSO_4	4.58% MnO.
	fusion in Ni crucible.	4.55% MnO.
Franklinite	Standard Method.	7.89% MnO.
	HNO_3 + HCl.	7.96% MnO.
Franklinite	Na_2O_2, iron pyrites, Mg,	7.90% MnO.
	KSO_4 in Ni crucible.	7.88% MnO.
Ferro-manganese	Standard Method. HNO_3	56.25% Mn.
		56.23% Mn.
Ferro-manganese	Na_2O_2, ZnS, KSO_4	56.18% Mn.
	in Ni crucible.	56.32% Mn.

The manganese in each case was determined by Volhard's method.

(m) The application of the sodium peroxide method of decomposition to silica determinations.

In order to investigate the adaptability of the sodium peroxide method to silica determinations, a blast furnace slag was decomposed by this method and by the standard method and the percentages of SiO_2 obtained by the two methods compared. For the standard method 1.0 gm. of the slag (200 mesh sieve) was boiled for an hour with hydrochloric acid (1.12 sp. gv.). The slag appeared to be completely decomposed. It was evaporated to dryness on the water bath and kept there for several hours after it was dry. It was moistened with hydrochloric acid, dissolved in water, and the silica filtered off. The filtrate was dehydrated a second time. The silica was ignited to constant weight in a platinum crucible and then volatilized with hydro-fluoric acid in the presence of a little sulphuric acid. The silica was determined by difference. The results obtained were 30.06% and 30.04% SiO_2.

Experiment No. 23.

8.0 gm. sodium peroxide.

1.2 gm. zinc sulphide.

0.3 gm. KSO_4.

0.5 gm. slag (200 mesh sieve).

Ignited the above mixture by means of magnesium ribbon in a nickel crucible. A good fusion was obtained.

The melt dissolved easily in water and no residue was left on acidifying with hydrochloric acid (1.12 sp. gv.). The silica was dehydrated twice and determined as before. The results were 30.02% and 29.98% SiO_2 as against 30.04% and 30.06% by the standard method.

(n) Some qualitative experiments with the sodium peroxide method.

Qualitative experiments were run on a number of substances to see if the sodium peroxide method would decompose them.

1. Experiments with chrome iron ore.

A very refractory chrome iron ore, high in iron and chromium was used.

Experiment No. 24.

8.0 gm. sodium peroxide.

1.5 gm. zinc sulphide.

0.5 gm chrome iron ore (200 mesh sieve).

0.3 gm. KSO_4.

Above mixture was ignited in a nickel crucible by means of magnesium ribbon. It was a good liquid fusion but the decomposition was incomplete. It was apparent that a zinc sulphide combustion did not produce heat enough, so a pyrite mixture was tried.

Experiment No. 25.

8.0 gm. sodium peroxide.

0.3 gm. powdered magnesium.

0.3 gm. KSO_4.

0.5 gm. Chrome iron ore (200 mesh sieve).

1.5 gm. pyrite.

Ignited above mixture by means of magnesium ribbon. The fusion was very hot. Sifted 3 to 4 gm. sodium peroxide into the liquid fusion to make it dissolve easily. The melt dissolved in sulphuric acid without leaving a residue.

2. Experiments with Monazite.

Monazite contains several of the rare metals, particularly thorium. The sodium peroxide method of fusion was tried on a sample containing 2.75% ThO_2.

Experiment No. 26.

8.0 gm. sodium peroxide.

1.2 gm. zinc sulphide.

1.0 gm. monazite (200 mesh sieve).

0.3 gm. KSO_4.

Ignited above mixture with magnesium ribbon. The fusion was good with no spattering. The decomposition was complete, for upon acidifying with hydrochloric acid no residue was left.

3. Experiments with clays.

A brick clay used was of the following composition: SiO_2 60.93%, Al_2O_3 17.93%, Fe_2O_3 8.12%, MgO 0.91%, CaO 1.33%, Na_2O and K_2O 5.01%, Vol. matter 5.73%. (This analysis and the one of the fire clay were made by the chemists of the Illinois State Geological Survey).

Experiment No. 27.

8.0 gm. sodium peroxide.

1.2 gm. zinc sulphide.

0.3 gm. KSO_4.

1.0 gm. clay (200 mesh sieve).

Ignited by means of a magnesium ribbon. The fusion was quiet. The fused mass dissolved completely in hydrochloric acid, leaving no residue.

Experiments were made with a fire clay of the following composition: SiO_2 67.37%, Al_2O_3 21.69%, Fe_2O_3 1.59%, FiO_2 1.32%, loss on ignition 8.70%.

Experiment No. 28.

8.0 gm. sodium peroxide.

0.3 gm. KSO_4.

1.2 gm. Zinc sulphide.

1.0 gm. fire clay (200 mesh sieve).

The above mixture was ignited with magnesium ribbon. A good quiet fusion took place. The melt when taken up in

water and made acid with hydrochloric acid, went into solution completely, leaving no insoluble residue.

4. Experiments with a copper matte.

A copper matte, high in copper sulphide was used in the following experiments.

Experiment NO.29.

8.0 gm. sodium peroxide.

0.5 gm. zinc sulphide.

0.3 gm. KSO_4.

1.0 gm. copper matte (200 mesh sieve).

This mixture was ignited by means of a magnesium ribbon. The fusion became red hot and very liquid. It was necessary to sift 3 to 4 gms. of sodium peroxide into the molten mass to make it dissolve easily. The melt when taken up in water and acidified with hydrochloric acid, dissolved completely, leaving no residue.

5. Experiments with Bauxite.

A sample of bauxite containing a considerable amount of iron and titanium oxides was used for the following experiments.

Experiment No. 30.

8.0 gm. sodium peroxide.

1.2 gm. zinc sulphide.

0.3 gm. KSO_4.

0.5 gm. bauxite (200 mesh sieve).

Ignited by means of magnesium ribbon. The fusion was good but the decomposition of the ore very poor. The addition of varying quantities of potassium chlorate did not produce decompositions which were much better. The pyrites mixture was tried.

Experiment No. 31.

8.0 gm. sodium peroxide.

0.5 gm. zinc sulphide.

0.5 gm. bauxite (200 mesh sieve).

1.0 gm. pyrites.

Ignited the above mixture by means of a magnesium ribbon. The fusion was extremely hot. 3 to 4 gms. of sodium peroxide were sifted into it to make the melt dissolve easily. The melt was dissolved in water and acidified with hydrochloric acid. No residue was left, which indicated complete decomposition. This method of decomposition is of no great importance for analytical purposes because of the introduction of iron.

6. Experiments with titanium iron ore.

A titanium iron ore, high in iron and titanium and very basic, was experimented with. A fusion of a mixture essentially the same as that used in experiment No. 30 did not decompose 0.5 gm. of the ore (200 mesh sieve).

A fusion of a mixture similar to that used in experiment No. 31 completely decomposed 0.5 gm. of the ore (200 mesh sieve). This also is of minor importance for analytical purposes because of the difficulty of determining titanium gravimetrically in the presence of large quantities of iron.

(o) Notes and precautions.

To ascertain the effect of the fusions on the nickel crucibles, two new crucibles were weighed and after sixteen fusions had been made in each, they were carefully cleaned and weighed again. A crucible weighing originally 18.7683 gm. lost .2876 gm. in 16 fusions. A crucible weighing originally 18.6119 gm. lost .3363 gm. in 16 fusions. This is an average loss in weight of about .02 gm. for each fusion, or about 0.10% of the weight of the crucible. When the hot, oxidizing nature of the fusions is considered, this is a very slight loss. The fusions were those made with the lead ores, using sodium peroxide and zinc sulphide.

It was found that a piece of twine about an inch long soaked in alcohol and lighted can be used for igniting the mixtures instead of magnesium ribbon. This could be used if for any reason magnesium was not to be introduced into the mixture.

A finely divided sample was found to be necessary for the sodium peroxide fusions. In the foregoing experiments, with the exception of the ferro-manganese and the lead

slag, it was found necessary to pass the substance to be analized through a 200 mesh sieve.

Since sodium peroxide often contains small lumps which keep the mixture from igniting or cause incomplete combustion, it is necessary to pulverize this substance before using it.

The different ingredients must be very thoroughly mixed. A partial mixing results in partial decomposition.

All the ingredients must be perfectly dry. The mixture is so easily ignited that if there is any moisture present, it may react with the sodium peroxide and evolve enough heat to ignite the mixture prematurely.

Conclusion.

From the foregoing experiments it is evident that sodium peroxide, zinc sulphide, and potassium persulphate, when mixed in certain proportions give a good fusion medium for the decomposition of the substances used in this investigation. In every case the new method gave results as good as those obtained by the standard method. With possibly slight modifications, it could be extended to numerous other substances, a few of which are indicated in the qualitative experiments. This method is particularly applicable to sulphide ores and to substances high in silica, such as glazes and slags. With basic substances such as franklinite and chrome iron ore it is necessary to use iron pyrite to get

sufficient heat. Pyrite, however, can not be used in a good many fusions where iron is objectionable, and to fusions of that nature this method can not be applied.

The chief advantage of the method is the quickness with which decomposition can be accomplished. The entire operation including mixing, igniting, cooling, and dissolving the melt, takes approximately five minutes. A decomposition by acid takes from half an hour to an hour and a half or even two hours. Moreover, in most cases the nitric or hydrochloric acid must be expelled by evaporating the solution down to sulphuric acid fumes, which necessitates considerable care and usually takes half an hour longer. In the sodium peroxide method sulphuric acid may be used directly for acidifying, which does away with the tedious evaporation.

In the new method the silica present is practically all brought into solution and kept there so that it does not clog filters and cause other annoyances. The sulphur in sulphide ores and the carbonaceous matter in slags, etc., is easily and completely oxidized by the sodium peroxide method and the laborious oxidation by means of nitric acid is avoided.

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